Measuring Particle Size Distribution Using Laser Diffraction: Implications for Predicting Soil Hydraulic Properties

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Abstract: Methods to predict soil hydraulic properties frequently require information on the particle size distribution (PSD). The objectives of this study were to investigate various protocols for rapidly measuring PSD using the laser diffraction technique, compare the obtained PSD with those determined using the traditional hydrometer-and-sieves method (HSM), and assess the accuracy of soil hydraulic properties predicted from the measured PSD. Ten soil samples encompassing a wide textural range were analyzed using the HSM and 3 different laser diffraction methods (LDM1, LDM2, and LDM3). In LDM1, the soil sample was thoroughly mixed before analysis. In LDM2, the sand fraction was sieved out and analyzed separately from the silt-clay fraction. LDM3 was similar to LDM2 except that the silt-clay fraction was diluted so that a large sample volume could be used while maintaining an acceptable level of obscuration. LDM2 and LDM3 improved the agreement between the PSD with the HSM in comparison to LDM1, without the need of altering the Mie theory parameters or the use of scaling factors. Moreover, a reasonable prediction of measured saturated hydraulic conductivity and water retention curve was achieved when using the PSD from LDM2 and LDM3, in conjunction with bulk density information.

Key words: Particle size distribution, laser diffraction, soil hydraulic properties.

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Analytical and numerical models (Philip, 1969; van Genuchtan and Wierenga, 1977; Šimůnek et al., 1999; Bradford et al., 2003) that simulate the transport and fate of water, solutes, organic compounds, and microorganisms in the vadose zone require as input information about soil hydraulic properties. Direct measurement of these properties (Klute, 1986; Klute and Dirksen, 1986) is laborious and expensive, especially when studying environmental processes at the farm, basin, or regional scales where numerous samples are needed (Hopmans et al., 2002; Vereecken et al., 2007).

Measurements of soil bulk density (ρ_b), particle size distribution (PSD), and soil texture (Blake and Hartage, 1986; Gee and Or, 2002) is usually simpler and less time consuming than direct measurements of water flow and retention. Methodologies have therefore been developed to estimate soil hydraulic properties from this information (Arya and Paris, 1981; Vereecken et al., 1990; Rajkai and Varallyay, 1992). In particular, pedotransfer functions based on large soil databases have been developed to predict soil hydraulic properties from more easily obtained soil physical data (Tietje and Tapkenhinrichs, 1993; Schaap and Leij, 1998; Schaap et al., 2001). Most of the clay, silt, and sand size fraction contained in those databases were obtained using sieving in combination with either the hydrometer or pipette method (e.g., Schaap et al., 2001).

The light scattering method has been used during the last decade to measure the PSD and the surface area of soils (Buurman et al., 1997; Eshel et al., 2004; Sperazza et al., 2004, Arriaga et al., 2006). Modern light scattering methods use polarization intensity differential scattering technology to study patterns of light (tungsten-halogen) and laser (780 nm) scattering in soil particle suspensions, which can be described mathematically by the Mie theory (Eshel et al., 2004; Arriaga et al., 2006; Berger et al., 2008). The advantages and disadvantages of these methods are well documented (Beuselinck et al., 1998; Eshel et al., 2004; Arriaga et al., 2006; Berger et al., 2008). Potential advantages of laser diffraction methods (LDM) include simplicity, small sample size, and rapid analysis and information on the full range of the PSD. Potential disadvantages include the need to make assumptions about particle geometry (sphere) and refractive indexes, overestimation of certain size categories because of competition between two light sources (Berger et al., 2008), a lack of LDM-determined PSD databases that can be used for comparison purposes, and a required initial expensive investment in equipment (Arriaga et al., 2006). To date, few studies have compared PSD information from LDM with PSD information obtained with traditional hydrometer-and-sieves method (HSM) (Konert and Vandenberghe, 1997; Beuselinck et al., 1998; Eshel et al., 2004; Berger et al., 2008).

Our research objectives were: (i) to investigate the agreement between PSD measured with laser diffraction and hydrometer methods, (ii) to predict hydraulic properties from PSD and bulk density measurements, and (iii) to evaluate the accuracy of the hydraulic properties predicted from laser diffraction–based PSD.

MATERIALS AND METHODS

We used eight pairs of undisturbed soil cores sampled from heterogeneous soil profiles in a field located near San Jacinto, CA (33°50′32″N, 117°00′30″W), and two disturbed soil samples from a field located west of Firebaugh, CA (36°49′44″N, 120°33′17″W) (Table 1). The undisturbed cores were obtained by driving brass cylinders measuring 6 cm tall and 5.4 cm in diameter into an exposed soil profile at variable depths. The two disturbed soil samples were air-dried, ground, passed through a 2-mm sieve, and packed in brass cylinders to their original bulk density (1.25 g·cm^{-3}). One core from each undisturbed pair was used to determine the ρ_b (Blake and Hartage, 1986). The eight remaining undisturbed cores and the two repacked cores were then used for measurement of the primary drainage branch of the water retention curve (WRC) and the saturated hydraulic conductivity (K_s), as later described. The samples were then used for PSD analysis by the HSM and LDM, also later described.

Particle Size Distribution

Sample preparation for both HSM and LDM was similar, except that 40 g of soil was used for the HSM and 4 g for LDM

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TABLE 1. Texture, Bulk Density, and Sand, Silt, and Clay Fractions of the 10 Soils

	Soil Separates, %				Bulk
Soil ID	Sand	Silt	Clay	Texture	Density, g cm^{-1}
Sa	93.64	3.86	2.50	Sand	1.45
LSa	75.95	16.55	7.50	Loamy sand	1.57
SaL-1	52.40	36.35	11.25	Sandy loam	1.35
SaL-2	55.51	35.25	9.24	Sandy loam	1.35
L-1	48.82	40.30	10.88	Loam	1.25
L-2	48.32	43.66	8.02	Loam	1.36
SiL-1	29.76	60.43	9.81	Silt loam	1.19
SiL-2	30.89	52.32	16.78	Silt loam	1.29
CL	26.47	35.05	38.48	Clay loam	1.25
SiC	15.11	40.87	44.02	Silty clay	1.25

(Gee and Or, 2002). Soil samples were ground, sieved (<2 mm), and oven dried (105 °C) overnight. After cooling down, 5 mL of 0.02 *M* NaCl was added per gram of soil and shaken for 1 h. The soil suspension was then centrifuged on 850 *g* for 20 min. After decanting, the samples were shaken overnight with 5 mL of sodium hexametaphosphate (NaHMP) solution per gram of soil.

The HSM was applied to soil suspensions following the protocol of Gee and Or (2002). In brief, hydrometer (VWR Scientific, West Chester, PA) measurements were made on the soil suspensions at specific times to determine the fine particle fractions. The soil suspensions were subsequently wet sieved (65, 105, 250, 500, and 1000 μm spacing size) to determine the coarser fractions. The weight basis PSD information from the HSM is equal to volume basis if constant soil particle density is assumed (2.65 g·cm^{-3}). For LDM, a laser diffractometry device (LA 930; Horiba LTD, Kyoto, Japan) was used to determine the PSD according to the Mie theory. Three different LDM protocols were considered, identified as LDM1, LDM2, and LDM3. In LDM1, 1 mL of well-mixed soil suspension was analyzed with the real and imaginary parts of the refractive index set equal to 1.44 and 0.2, respectively (Eshel et al., 2004). In LDM2 and LDM3, wet sieving with a 50-μm sieve was used to separate the soil suspension into two size fractions, sand and silt-clay. A subsample (0.5 mL) of the silt-clay suspension was analyzed immediately with the laser diffractometer using values of 1.5 and 0.1 for the real and imaginary parts of the refractive index, respectively, which are considered to be more appropriate values when analyzing samples with only finer-sized particles (Eshel et al., 2004). In contrast, the sand fraction (>50 μm) was oven dried (105 °C) overnight and weighed. Any remaining organic matter was removed before analysis. The sand was then resuspended in the NaHMP solution, equilibrated for 1 h, and analyzed with the laser diffractometer (using the same refractive indices used in LDM1). The PSD of the silt-clay and sand analyses, made with the laser system, were merged based on the relative weight of each fraction.

The obscuration levels of samples in the laser diffractometry analysis were kept between 7 % and 13 %. Maintaining this obscuration levels in soils with high clay contents (>20 %) compelled us to use small volumes because of the high optical density of clay. In LDM3, the silt-clay fraction was diluted with NaHMP (1:10 ratio) so that the same volume of suspension could be used but containing a lower concentration of particles.

Measured and Predicted Soil Hydraulic Properties

A constant head permeameter (Klute and Dirksen, 1986) was used to measure saturated hydraulic conductivity (K_s) on each core. A standard solution (calcium chloride, 0.003 *M*) was allowed to infiltrate for 24 h at a head of 5 cm. After reaching steady state, the flow rate was determined by weighing the effluent over a known time interval.

The K_s was predicted based on the Kozeny-Carmen model (Lagerwerff et al., 1969), which relates the particle surface area to water permeability and hence K_s. The geometrical surface area per particle volume (cm^2·cm^{-3}) was calculated from the PSD information (e.g., Jury et al., 1991):

$$A_s = 6 \cdot 10^4 \times \sum_{i=1}^{i=j} \frac{F_i}{D_i} \tag{1}$$

where F_i is the frequency for the *i*th particle size fraction, and D_i is the mean particle diameter of the *i*th particle size fraction (μm). This quantity can be converted to units of square centimeters per gram by dividing by ρ_b.

The primary drainage branch of the WRC from each undisturbed core (between −1 and −800 cm of pressure head) was measured using Tempe cells and the multistep outflow technique (Klute, 1986; Eching et al., 1994). A Tempe cell apparatus was used to control and record 10 outflow cells that were run in parallel. The water contents at lower water pressure heads (between −1,000 and −15,000 cm) were measured using a pressure

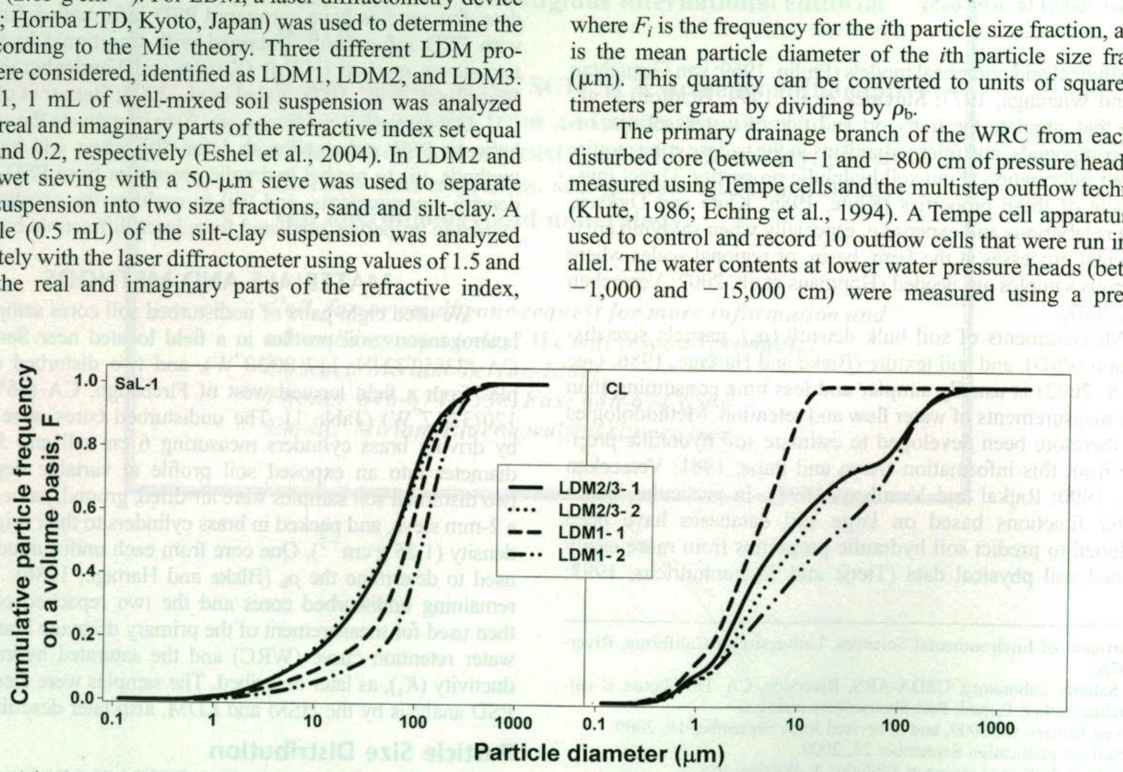

FIG. 1. Particle size distribution of sandy loam and clay loam soil, as generated by the LDM. LDM1, LDM2, and LDM3 are three preparation protocols of soil sample before the analysis with the laser method.

plate apparatus (Soilmoisture Equip. Corp, Santa Barbara, CA) according to the approach outlined by Richards (1965). The measured WRC was parameterized for subsequent analysis using the model of van Genuchten (1980).

The Arya-Paris model (Arya and Paris, 1981), which assumes the pore size distribution curve mimics the soil WRC, was used to predict the relationship among PSD, ρ_b, and the WRC. The soil coefficient of the Arya-Paris model was taken to be 1.285 for sand, 1.459 for sandy loam, 1.375 for loam, and 1.15 for silt-loam (Arya and Paris, 1981). Because of the discrete form of the PSD data, the predicted WRC from the Arya and Paris model were also discrete. Parameters from the van Genuchten (1980) model were fitted to these retention data sets so that the agreement between measured and predicted data could be better quantified.

Additional predictions of soil hydraulic properties were obtained using the Rosetta pedotransfer function model, which can predict soil hydraulic properties (K_s and the WRC model parameters) from ρ_b and the percentages of sand, silt, and clay (Schaap et al., 2001).

Statistical analysis was performed on the measured PSD data to assess the agreement of LDM1, LDM2, and LDM3 methods with the conventional HSM method and to quantify the agreement of observed and predicted hydraulic properties (K_s, A_s, and WRC). Statistical measures of goodness of fit

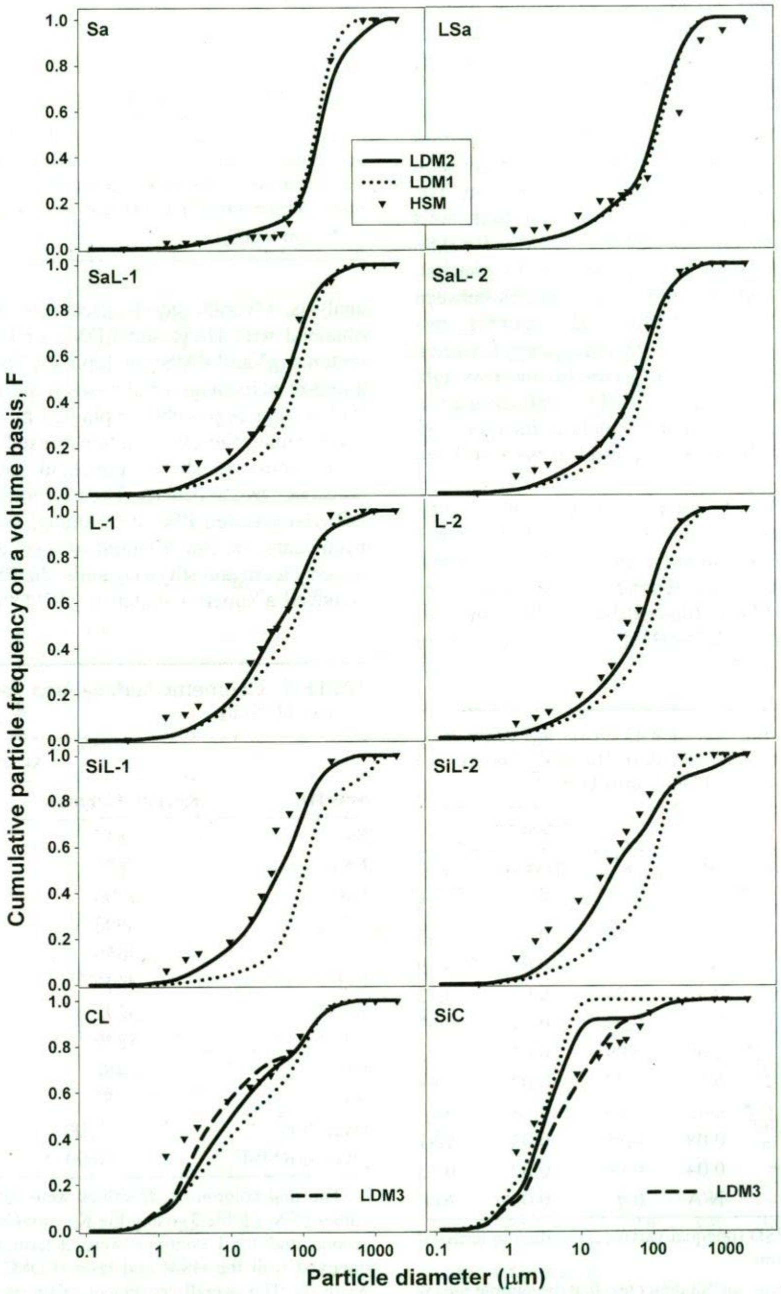

FIG. 2. Particle size distributions for the 10 soils by the HSM and LDM. LDM1, LDM2, and LDM3 are three preparation protocols of soil sample before the analysis with the laser method.

included the coefficient of linear regression (r^2), the relative mean square error (RMSE), and a two-tailed Student t test with unequal variance.

RESULTS AND DISCUSSION

Particle Size Distribution

Bulk density, texture, and sand, silt, and clay fractions obtained with the HSM are presented in Table 1. The soils encompass a wide range of texture classes, from sand to silty clay. The soil separate percentages ranged from 15 % to 93 % for sand, 3 % to 60 % for silt, and 2.5 % to 44 % for clay. Bulk density ranged from 1.19 to 1.57 g·cm^{-3}.

Figure 1 presents example particle size distributions measured with the LDM. Figure 1 shows the cumulative particle frequency (F) on a volume basis plotted versus particle diameter as measured by the laser method for sandy loam (SaL-1) and clay loam (CL) soils. The dashed and dash-dotted lines are two repetitions of LDM1, in which all the size fractions were simultaneously analyzed. The solid and dotted lines are data from two repetitions of LDM2 or LDM3 (LDM2/3), where the sand fraction was separately analyzed from the silt-clay fraction. The agreement between the dotted and solid lines for both soils demonstrates the good repeatability of LDM2 or LDM3 ($P = 0.88$ and $P = 0.94$ for SaL-1 and CL, respectively). In contrast, duplicate samples from LDM1 showed inconsistencies between the repetitions ($P = 0.36$ and $P = 0.00$ for SaL-1 and CL, respectively). One potential explanation for this inaccuracy is caused by competition of the various size categories for the two light sources. This competition is especially likely to influence measurements of the size class having the smallest fractions; for example, clay in the sandy loam and the sand in the clay loam (Table 1).

The cumulative particle frequency (F) by volume versus particle diameter measured by the HSM (triangles), LDM1 (dotted line), and LDM2 (solid line) for all 10 soils is presented in Fig. 2. Also shown are curves generated for the clay loam and silty clay soils using LDM3 (dashed lines), where the silt-clay fraction was diluted with NaHMP (1:10 ratio) before analysis. Overall, good agreement was found between PSD obtained with HSM and LDM2 or LDM3 (LDM2/3) over all textures (r^2 and RMSE in Table 2). The HSM tended to indicate a higher percentage of clay-sized particles than did LDM2/3, a finding that is possibly explained by the tendency of HSM to overestimate the clay fraction caused by the assumptions of a single spherical shape (Loveland and Whalley, 1991) and a constant particle density. The agreement between the HSM- and LDM1-measured PSD deteriorated as the samples transitioned from coarse- to fine-textured soils (r^2 and RMSE in Table 2). In the clay loam and silty clay soils, the PSD obtained using LDM3 achieved a superior match to HSM than LDM2 (Fig. 2). This

TABLE 2. Statistical Comparison of PSD Obtained With the HSM Versus Distributions Obtained With the LDM Using Different Preparation Protocols LDM1 and LDM2/3

	LDM1			LDM2/3		
Soil ID	r^2	RMSE	P*	r^2	RMSE	P*
Sa	1.00	0.02	0.93	0.99	0.02	0.73
LSa	0.97	0.02	0.48	0.96	0.03	0.45
SaL-1	0.97	0.05	0.79	0.99	0.01	0.99
SaL-2	0.94	0.08	0.58	0.99	0.01	0.83
L-1	0.99	0.03	0.69	0.99	0.01	0.67
L-2	0.97	0.04	0.69	0.99	0.01	0.93
SiL-1	0.82	0.14	0.10	0.97	0.02	0.68
SiL-2	0.87	0.12	0.42	0.99	0.03	0.17
CL	0.64	0.07	0.08	0.95	0.03	0.91
SiC	0.80	0.03	0.04	0.96	0.02	0.83
Overall	0.86	0.06	N/A	0.97	0.02	N/A

*t test conducted on the PSD (unequal variances) written in terms of the probability density function.

P: probability associated with the Student t test that the sample means are equal; r^2: coefficient of linear correlation.

TABLE 3. Measured and Predicted K_s for the 10 Soils

	K_s, cm h^{-1}			
Soil ID	Measured	Rosetta	HSM	LDM2/3
Sa	32.34	31.04	63.22	20.50
LSa	1.73	2.75	4.63	1.96
SaL-1	2.34	1.78	5.33	1.62
SaL-2	1.28	2.25	8.65	2.00
L-1	0.50	2.23	7.43	1.97
L-2	0.61	1.75	8.70	1.72
SiL-1	2.07	3.50	14.01	2.61
SiL-2	0.51	1.23	3.35	0.89
CL	0.30	0.98	0.57	0.16
SiC	0.06	0.92	0.73	0.08
Overall r^2*	1.00	0.82	0.78	0.85
Overall RMSE	0.00	2.50	36.99	1.18

The HSM and laser (LDM2 and LDM3) predictions were based on the geometric surface area calculation, whereas the Rosetta prediction was obtained by the pedotransfer function. The overall coefficient of linear correlation (r^2) and RMSE are also provided.

*r^2 on log(K_s).

TABLE 4. Geometric Surface Area Per Particle Volume (A_s) for the 10 Soils

	As, cm^2·cm^{-3}		
Soil ID	Kozeny-Carman	HSM	LDM2/3
Sa	815	584	1026
LSa	2813	1724	2646
SaL-1	3700	2455	4454
SaL-2	4992	1926	4009
L-1	9659	2509	4867
L-2	7079	1885	4239
SiL-1	5292	2044	4734
SiL-2	8859	3466	6711
CL	12,486	8042	17,053
SiC	27,781	9071	23,210
Overall r^2	1.00	0.79	0.87
Overall RMSE	0.00	3135	693

The first column of A_s values were calculated from the measured values of K_s (Table 3) using the Kozeny-Carman equation, whereas the second and third columns were determined from PSD information obtained with the HSM and laser (LDM2 and LDM3) methods, respectively. The overall coefficient of linear correlation (r^2) and RMSE are also provided.

might be caused by the high optical density associated with high silt-clay fractions and the fact that less than 0.1 mL of sample was needed to obtain the correct level of obscuration when using LDM2. This diminutive volume may not be a representative sample and can induce variations. The dilution (LDM3) allows analyzing a larger volume of silt-clay suspension while maintaining an acceptable level of obscuration.

Soil Hydraulic Properties

This section compares measured and predicted soil hydraulic and physical properties (K_s, A_s, and WRC) for the 10 soils. Predictions were obtained using PSD information from HSM and LDM2/3 methods and/or the Rosetta pedotransfer function. Information on the PSD from LDM1 was not considered in this analysis because of the limitations previously noted.

Measured and predicted K_s values for the soils are presented in Table 3. The measured K_s values ranged over three orders of magnitude. All three methods (HSM, LDM2/3, and Rosetta) provided a relatively good estimation of K_s, with values of r^2 on $\log(K_s)$ values ranging from 0.78 to 0.85. However, the overall prediction by LDM2/3 was found to be better than others (smallest RMSE and the highest r^2 in Table 3).

The geometric surface area per particle volume (A_s) for the various soils was determined from the PSD of HSM and LDM2/3 analysis methods, and this information is presented in Table 4. For comparison purposes, A_s was also calculated from

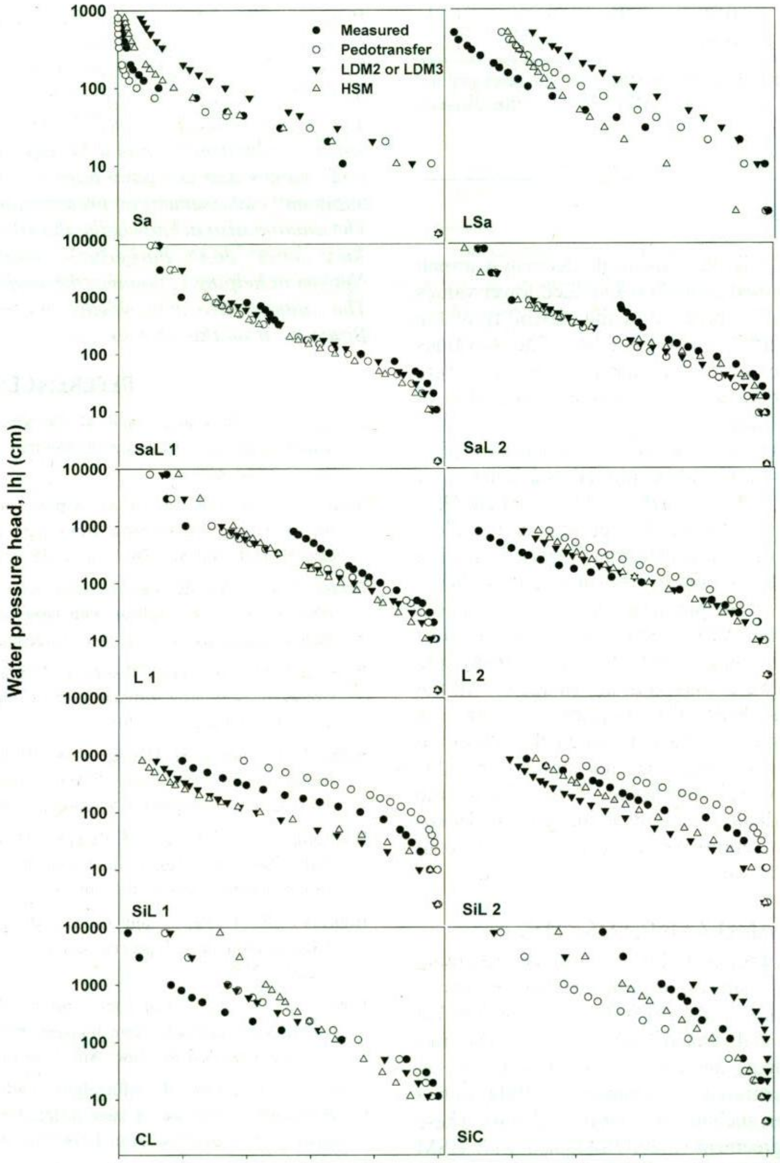

FIG. 3. Measured and predicted WRC of the 10 soils. Here, water pressure head (h) is plotted as a function of the effective water saturation (S_e). Predicted WRC used PSD obtained from the HSM, LDM (LDM2 or LDM3), and the Rosetta pedotransfer function.

TABLE 5. Statistical Comparison of the Measured and Predicted WRC

	Rosetta		HSM		LDM2/3	
Soil ID	r^2	RMSE	r^2	RMSE	r^2	RMSE
Sa	0.95	0.03	0.98	0.04	0.96	0.59
LSa	0.98	0.31	0.92	0.33	0.93	0.94
SaL-1	0.97	0.01	0.97	0.01	0.97	0.01
SaL-2	0.91	0.04	0.93	0.03	0.93	0.03
L-1	0.92	0.02	0.90	0.03	0.92	0.02
L-2	0.98	0.15	0.99	0.07	0.99	0.05
SiL-1	0.94	0.10	0.91	0.09	0.88	0.07
SiL-2	0.94	0.04	0.97	0.01	0.94	0.04
CL	0.97	0.17	0.96	0.81	0.98	0.04
SiC	0.97	0.08	0.99	0.01	0.79	0.19
Overall	0.80	0.09	0.84	0.14	0.77	0.06

Predictions were based on PSD from the HSM and the laser method with different preparation protocols (LDM2/3) and the Rosetta pedotransfer function.

r^2: coefficient of linear correlation.

the measured values of K_s (Table 3) using the Kozeny-Carmen equation. The PSD determined using HSM yielded lower values of A_s relative to the LDM2/3 because of the capability of the LDM to detect smaller particles than the HSM. The A_s values from LDM2/3 were closer to the calculated values that were determined from measured values of K_s, having an overall r^2 of 0.87 and a lower RMSE (Table 4).

Measured and predicted WRC for the soils are presented in Fig. 3, with the water pressure head (h) plotted as a function of the effective saturation, $S_e = (\Theta - \Theta_r)\cdot(\Theta_s - \Theta_r)^{-1}$, where Θ, is the water content, and Θ_s and Θ_r are the saturated and residual water contents. The value of Θ_r was determined by optimization of the van Genuchten (1980) model to measured and predicted WRC data. In general, all of the prediction methods provided a reasonable description of the WRC when written in terms of S_e (r^2 and RMSE, Table 5). No single method (Rosetta, HSM, and LDM2/3) was found to have a consistent advantage for all the soils (Table 5). When the WRC data is plotted in terms of saturation (Θ/Θ_s), then the agreement between the measured and predicted WRC is much lower (data not shown), and this indicates sensitivity to the selected value of Θ_r. Hence, the accuracy of the WRC prediction methods is likely to be dependent on differences in the optimized value of Θ_r. Additional research is warranted on this topic.

SUMMARY AND CONCLUSIONS

The particle size distribution (PSD) of 10 soils representing a wide textural range was analyzed by four different protocols: the traditional HSM, a mixed LDM (LDM1), a separated LDM (LDM2), and a separated and diluted LDM (LDM3). The new LDM2 and LDM3 protocols analyzed the sand and silt-clay fraction separately and permitted measurement of PSD with a wide range of particle sizes, such as found in natural soils. These protocols improved the agreement of PSD obtained with HSM and LDM without the need of altering the Mie theory parameters or the use of scaling factors. Moreover, PSD obtained with LDM2 were found to be superior to the alternative methods (i.e., HSM PSD and a pedotransfer function) for predicting K_s. A satisfactory prediction of the water retention curve was also found for a wide textural range of soils when using PSD from HSM and LDM2/3 methods, provided the data were plotted in terms of effective saturation. Moreover, prediction accuracy was found to be equivalent to alternative tools, such as pedotransfer functions. The integrated benefits of the separated protocols and the laser diffraction technique will facilitate its use in studies requiring analysis of PSD on numerous soils sample (i.e., large areas).

ABBREVIATIONS

PSD: particle size distribution;
HSM: hydrometer and sieves method;
LDM: laser diffraction method;
WRC: water retention curve.

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